



INVESTOR IN PEOPLE

The Patent Office  
 Concept House  
 Cardiff Road  
 Newport  
 South Wales  
 NP10 8QQ

FILED 19 DEC 2003

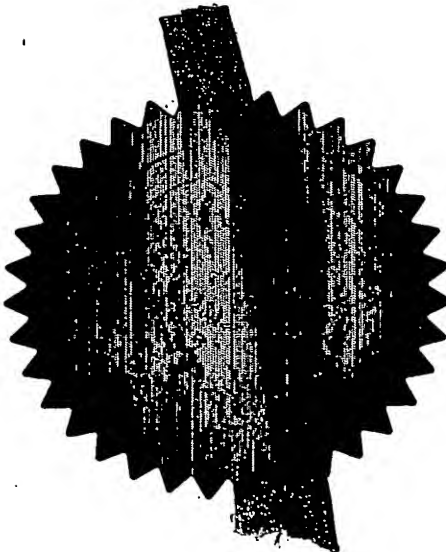
WFO PCT

I, the undersigned, being an officer duly authorised in accordance with Section 74(1) and (4) of the Deregulation & Contracting Out Act 1994, to sign and issue certificates on behalf of the Comptroller-General, hereby certify that annexed hereto is a true copy of the documents as originally filed in connection with the patent application identified therein.

In accordance with the Patents (Companies Re-registration) Rules 1982, if a company named in this certificate and any accompanying documents has re-registered under the Companies Act 1980 with the same name as that with which it was registered immediately before re-registration save for the substitution as, or inclusion as, the last part of the name of the words "public limited company" or their equivalents in Welsh, references to the name of the company in this certificate and any accompanying documents shall be treated as references to the name with which it is so re-registered.

In accordance with the rules, the words "public limited company" may be replaced by p.l.c., plc, P.L.C. or PLC.

Re-registration under the Companies Act does not constitute a new legal entity but merely subjects the company to certain additional company law rules.



Signed

*R. Mahoney*

Dated 7 November 2003

**PRIORITY  
 DOCUMENT**

SUBMITTED OR TRANSMITTED IN  
 COMPLIANCE WITH RULE 17.1(a) OR (b)

**BEST AVAILABLE COPY**

An Executive Agency of the Department of Trade and Industry

# Patents Form 1/77

Patents Act 1977  
(Rule 1)



1/77

The Patent Office

Cardiff Road

Newport  
NP23 1RH

## Request for grant of a patent

(See the notes on the back of this form. You can also get an explanatory leaflet, from the Patent Office to help you fill in this form)

1.	Your reference	CDK 2045	08NOV02 E762121-9 D02806
2.	Patent application number (The Patent Office will fill in this part)	0226101.4	FOI/7700 0.00-0226101.4
3.	Full name, address and postcode of the or of each applicant ( <u>underline all surnames</u> )	Rhodia Consumer Specialties Limited Trinity Street Oldbury West Midlands B69 4LN	
	Patents ADP number (if you know it)	7870322005	
	If the applicant is a corporate body, give the country/state of its incorporation	England	
4.	Title of the invention	WHITE RUST CORROSION INHIBITORS	
5.	Name of your agent (if you have one)	Barker Brettell	
	"Address for service" in the United Kingdom to which all correspondence should be sent (including the postcode)	138 Hagley Road Edgbaston Birmingham B16 9PW	
	Patents ADP number (if you know it)	7442494002	
6.	If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and (if you know it) the or each application number	Country	Priority application number (if you know it)
7.	If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application	Number of earlier application	Date of filing (day/month/year)
8.	Is a statement of inventorship and of right to grant of a patent required in support of this request (Answer 'Yes' if: a) any applicant named in part 3 is not an inventor, or b) there is an inventor who is not named as an applicant, or c) any named applicant is a corporate body. See note (d))	YES	

Patents Form 1/77

# Patents Form 1/77

Enter the number of sheets for any of the following items you are filing with this form.  
Do not count copies of the same document

Continuation sheets of this form -

Description 9 + 9 ✓

Claim(s) 4 + 4 ✓

Abstract - *DM*

Drawing(s) -

0. If you are also filing any of the following, state how many against each item.

Priority documents -

Translations of priority documents -

Statement of inventorship and right to grant of a patent (*Patents Form 7/77*) -

Request for preliminary examination 1  
(*Patents Form 9/77*)

Request for substantive examination -  
(*Patents Form 10/77*)

Any other documents -  
(*please specify*)

1. I/We request the grant of a patent on the basis of this application.

Signature  
*Barker Brettell*  
Barker Brettell

Date  
07 November 2002

12. Name and daytime telephone number of person to contact in the United Kingdom

C D Kinton

Tel: 0121 456 1364

## Warning

After an application for a patent has been filed, the Comptroller of the Patent Office will consider whether publication or communication of the invention should be prohibited or restricted under Section 22 of the Patents Act 1977. You will be informed if it is necessary to prohibit or restrict your invention in this way. Furthermore, if you live in the United Kingdom, Section 23 of the Patents Act 1977 stops you from applying for a patent abroad without first getting written permission from the Patent Office unless an application has been filed at least 6 weeks beforehand in the United Kingdom for a patent for the same invention and either no direction prohibiting publication or communication has been given, or any such direction has been revoked.

## Notes

a) If you need help to fill in this form or you have any questions, please contact the Patent Office on 01645 500505

b) Write your answers in capital letters using black ink or you may type them.

c) If there is not enough space for all the relevant details on any part of this form, please continue on a separate sheet of paper and write "see continuation sheet" in the relevant part(s). Any continuation sheet should be attached to this form.

d) If you have answered 'Yes' Patents Form 7/77 will need to be filed.

e) Once you have filled in the form you must remember to sign and date it.

f) For details of the fee and ways to pay please contact the Patent Office.

Patents Form 1/77

## WHITE RUST CORROSION INHIBITORS

This invention relates to corrosion inhibitors and in particular to corrosion inhibitors for use in systems where water is used. The present invention especially relates to a compound for use as a white rust corrosion inhibitor and to a composition including such a compound.

Many of the components of water using systems are made of steel, which has been galvanised with zinc or cadmium by various processes, including hot dip. This is done in order to minimise corrosion of the steel. Zinc is the most commonly used metal for galvanising steel.

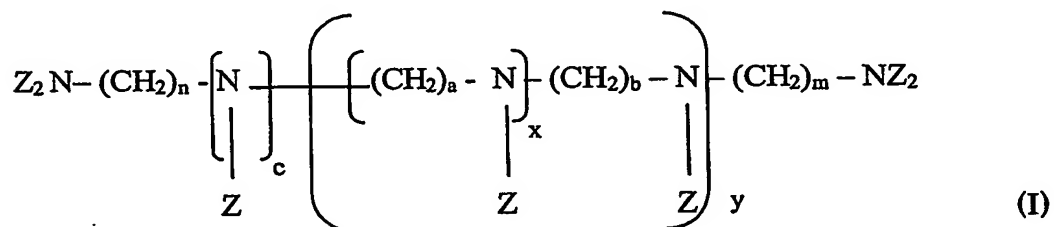
The zinc plating protects the steel from corrosion. However, the zinc plating reacts with the atmosphere and with water to produce "white rust" which comprises a variety of zinc compounds including, zinc oxide, zinc carbonates and the like. Under these corrosive conditions the zinc coating may not be able to protect the base steel and thus corrosion of the steel may occur reducing the life expectancy of the system. White rust is therefore problematic in water using industries utilising galvanised steel systems. This has been recognised in the industry (as is shown, for example in "White Rust: An Industry update and Guide Paper" published by the Association of Water Technologies, 2002). However, current philosophy in the industry is to concentrate on the treatment and removal of white rust after it has formed, and/or on its avoidance by selecting construction materials which are not susceptible to its formation.

Hitherto, no corrosion inhibitor currently used in water using systems has been found which is able to inhibit effectively the formation of "white rust".

It is an object of the present invention to provide a compound and a composition, each of which is effective for inhibiting "white rust" formation and for ameliorating the problem of corrosion caused by "white rust" in systems that have been galvanised with relatively electropositive metals such as zinc or cadmium, especially with zinc.

The applicant has found that the addition, to water using systems, of certain phosphonated oligomers or of random copolymers of vinylidene diphosphonic acid and vinyl sulphonic acid, or of compositions including such compounds, achieves the aforementioned object.

Accordingly, the present invention provides in a first aspect a compound for use as, or in connection with, a white rust corrosion inhibitor for water-treatment, said compound consisting of an organophosphonate having the general formula (I):



Wherein

$Z = -CHR_1PO_3R_2$

$R = H, CH_3, C_2H_5$  or  $M$

$R^1 = H, CH_3, CR_3, C_6H_5$ , or  $SO_3H_2$

$M =$  alkali metal or ammonium ion

$n = 0$  to  $6$

$m = 0$  to  $6$

$a = 0$  to  $6$

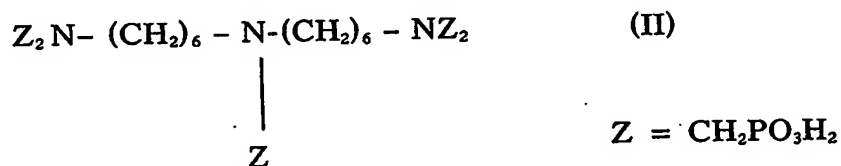
$b = 0$  to  $6$

$c = 0$  or  $1$

x = 0 to 6

y = 0 to 6

In a particularly preferred embodiment of the present invention R and R'  
 5 each = H, n = 6, m = 6, c = 1, y = 0 whereby the compound is  
 bis(hexamethylene)triamine-pentakis (methylene phosphonic acid), as in  
 formula (II):



10

In a second aspect, the present invention provides a compound for use as  
 a white rust corrosion inhibitor for water-treatment, said compound being  
 a random copolymer of vinylidene diphosphonic acid and vinyl sulphonic  
 15 acid in a molar ratio of between 1:1 and 1:500, suitably about 1:100  
 molar and preferably about 1:20 molar.

In a third aspect the present invention provides a composition for use as,  
 or in connection with a corrosion inhibitor for water-treatment, said  
 20 composition comprising a phosphonated oligomer according to the first  
 aspect or a random copolymer of vinylidene diphosphonic acid and vinyl  
 sulphonic acid, according to the second aspect, together with additives  
 conventionally used in the water treatment industry. The additives may  
 include scale inhibitors such as phosphonates, corrosion inhibitors such as  
 25 phosphonocarboxylic acids or salts and/or dispersants such as  
 polyacrylates. The composition may optionally incorporate a biocide.

In a fourth aspect, the present invention provides a method for inhibiting corrosion in, or in connection with, a water-using system, said method consisting of the application or addition to said system of an effective amount of a phosphonated oligomer according to the first aspect or a  
 5 random copolymer of vinylidene diphosphonic acid and vinyl sulphonic acid according to the second aspect or of a composition according to the third aspect.

Preferably, in the method according to the fourth aspect of the present  
 10 invention, the oligomer or co-polymer or composition is used in an effective amount of up to 1000 ppm suitably up to 250 ppm, for example up to 100 ppm

The phosphonocarboxylic acid or salt is preferably a phosphonated  
 15 oligomer of maleic acid, of general formula (III):



wherein M is a cation such that the oligomer is soluble in water, and n is  
 20 greater than 1. Such phosphonated oligomer's are disclosed in the applicant's EP-B-0491391 and equivalent publications and are available from the applicant as BRICORR® 288.

The polyacrylate compound is preferably a low molecular weight (MW)  
 25 polymer, e.g. MW = 2000-5000. However, it is to be understood that other molecular weight ranges can be used.

The present invention will now be illustrated, merely by way of example,  
 as follows.

## Methodology

### Test Water

Two test waters were used in the Examples herein below. The relevant properties of the test waters are shown in Table 1 (below).

**TABLE I**

	Water 1 (Soft)	Water 2 (Hard)
Calcium Hardness (ppm as $\text{CaCO}_3$ )	0	340
Alkalinity (ppm as $\text{CaCO}_3$ )	500	300
Chloride ion (ppm)	150	50
pH	9	9

The waters shown in TABLE 1 (above) are known to be corrosive to galvanised steel.

### Test Method

The corrosion testing was carried out on a Rotating Coupon Rig. Each Rig holds 2 coupons. The test water was aerated and the galvanised steel coupons (50 X 25mm) rotated at 150-160rpm. Water losses due to evaporation were replaced with de-ionised water daily.

All inhibitors used in the Examples (below) were weighed out on an analytical balance and added directly to the test water. The (pre-weighed) galvanised steel coupons were then rotated for 7 days at 40°C, cleaned to remove any corrosion deposits with warm (70-80°C) ammonium acetate

solution and then rinsed with water and acetone. The galvanised steel coupons were then oven dried and re-weighed.

#### **EXAMPLES A TO E**

- 5 The corrosion-inhibiting properties of six compounds or compositions according to the present invention were assessed by subjecting galvanised steel coupons to the test method described herein above. Also included for comparison are results from tests using phosphonates that are conventionally used as steel corrosion inhibitors (examples G, H and I).
- 10 The result are shown in TABLE II (below):

TABLE II

Inhibitor used (see notes below)	Inhibitor Level (ppm)	0 Ca <sup>++</sup> Water (Soft)	340 Ca <sup>++</sup> Water (Hard)
		Corrosion Rate (% of control)	Corrosion Rate (% of Control)
Control	0	100	100
Example (A)	100	20	59.2
Example (B)	100	12	89.2
Example (C)	100	15	93.3
Example (D)	100	14	115
Example (E)	100	20	44.2
Example (F)	100	-	51
Example (G)	100	196	131
Example (H)	100	112	-
Example (I)	100	82	-

## Notes to TABLE II

- 5 (A) a (random copolymer of Vinylidene Diphosphonic Acid (VDPA) and Vinyl Sulphonic Acid (VSA) in a 1:20 molar ratio) available from the applicant as ITC 1028;
- 10 (B) a 23% w/w neutral solution of the potassium salt of hexamethylene diamine tetrakis(methylenephosphonic acid), available from the applicant as BRIQUEST ® 462;
- (C) a 25% w/w, neutral, aqueous solution of a sodium salt of pentaethylenhexamine-octakis (methylenephosphonic acid), available from the applicant as BRIQUEST ® 8106-25S;
- 15 (D) a 30% w/w aqueous solution of a sodium salt of N, N-bis (3-aminopropyl)amine-hexakis (methylenephosphonic acid), available from the applicant as BRIQUEST ® 684-30S;
- 20 (E) a 40% w/w aqueous solution of bis(hexamethylene)triamine pentakis(methylenephosphonic acid), available from the applicant as BRIQUEST ® 5123-45A.
- 25 (F) a mixture of BRIQUEST ® 5123-45A with 100ppm of a formulation containing water treatment polymers and a phosphonated oligomer of maleic acid, available from the applicant as BRICORR ® 288C.
- (G) BRICORR ® 288C on its own.
- 30 (H) a 60% w/w aqueous solution of 1-hydroxyethane-1,1-diphosphonic acid available from the applicant as BRIQUEST ® ADPA-60A

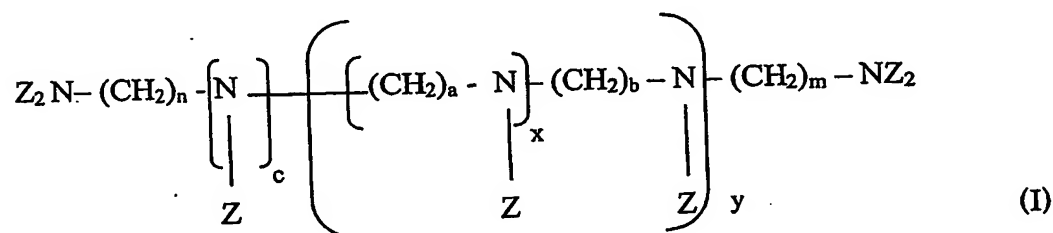
- (I) a 50% w/w aqueous solution of Nitrilotris(methylenephosphonic acid) available from the applicant as BRIQUEST® 301-50A

5 It will be apparent from TABLE I that inhibitor (E) gives the best all-round performance.

7rt80

## CLAIMS

1. A compound for use as, or in connection with, a white rust corrosion inhibitor for water-treatment, said compound consisting of an organophosphonate having the general formula (I):



Wherein

$Z = -CHR_1PO_3R_2$

$R = H, CH_3, C_2H_5$  or  $M$

10  $R^1 = H, CH_3, CR_3, C_6H_5$ , or  $SO_3H_2$

$M =$  alkali metal or ammonium ion

$n = 0$  to  $6$

$m = 0$  to  $6$

$a = 0$  to  $6$

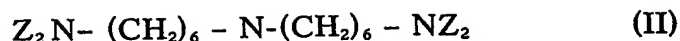
15  $b = 0$  to  $6$

$c = 0$  or  $1$

$x = 0$  to  $6$

$y = 0$  to  $6$

- 20 2. A compound as claimed in Claim 1, in which  $R$  and  $R^1$  each =  $H$ ,  $n = 6$ ,  $m = 6$ ,  $c = 1$ ,  $y = 0$  whereby the compound is bis(hexamethylene)triamine-pentakis (methylene phosphonic acid), as in formula (II):



3. A compound for use as a white rust corrosion inhibitor for water-treatment, said compound being a random copolymer of vinylidene diphosphonic acid and vinyl sulphonic acid in a molar ratio of  
5 between 1:1 and 1:500.
4. A compound as claimed in Claim 3, in which the molar ratio is 1:100 molar.
- 10 5. A compound as claimed in Claim 3 or Claim 4, in which the molar ratio is 1:20 molar.
6. A composition for use as, or in connection with a corrosion inhibitor for water-treatment, said composition comprising a  
15 phosphonated oligomer according to Claim 1 or a random copolymer of vinylidene diphosphonic acid and vinyl sulphonic acid, according to Claim 2, together with additives conventionally used in the water treatment industry.
- 20 7. A composition as claimed in Claim 6 in which the additives are selected from the group consisting of phosphonocarboxylic acids or salts and dispersants.
8. A composition as claimed in Claim 6 or Claim 7 in which the  
25 dispersant is a polyacrylate.

9. A composition as claimed in any one of Claims 6 to 8 in which the composition comprises a biocide.

10. A composition as claimed in any one of Claims 6 to 9 in which the  
5 phosphnocarboxylic acid or salt is a phosphonated oligomer of maleic acid, of general formula (III):



10 wherein M is a cation such that the oligomer is soluble in water, and n is greater than 1.

11. A composition as claimed in any one of Claims 6 to 9, in which the  
15 polyacrylate compound is a low molecular weight polymer having a molecular weight between 2000 to 5000.

12. A method for inhibiting corrosion in, or in connection with, a water-using system, said method consisting of the application or addition to said system of an effective amount of a phosphonated oligomer  
20 according to Claim 1 or a random copolymer of vinylidene diphosphonic acid and vinyl sulphonic acid according to Claim 2 or of a composition according to Claim 3.

13. A method as claimed in Claim 12, in which the oligomer or  
25 copolymer is used in an effective amount of up to 1000 ppm.

14. A method as claimed in Claim 12 or Claim 13, in which the oligomer or copolymer is used in an effective amount of up to 250 ppm.

30 15 A method as claimed in any one of Claims 12 to 14 in which the oligomer or copolymer is used in an effective amount of up to 100 ppm.

16. A compound for use as or in connection with, a white rust corrosion inhibitor substantially described herein with reference to the general formula.

5 17. A compound for use as a white rust corrosion inhibitor for water treatment substantially as described herein.

18. A composition for use as, or in connection with, a corrosion inhibitor for water treatment substantially as described herein.

10

19. A method for inhibiting corrosion, or in connection with a water-using system substantially as described herein.

**This Page is Inserted by IFW Indexing and Scanning  
Operations and is not part of the Official Record**

**BEST AVAILABLE IMAGES**

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

☒ BLACK BORDERS

☐ IMAGE CUT OFF AT TOP, BOTTOM OR SIDES

☒ FADED TEXT OR DRAWING

☐ BLURRED OR ILLEGIBLE TEXT OR DRAWING

☐ SKEWED/SLANTED IMAGES

☐ COLOR OR BLACK AND WHITE PHOTOGRAPHS

☐ GRAY SCALE DOCUMENTS

☐ LINES OR MARKS ON ORIGINAL DOCUMENT

☒ REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY

☐ OTHER: \_\_\_\_\_

**IMAGES ARE BEST AVAILABLE COPY.**

**As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.**